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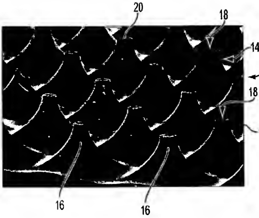
(54) **Title:** MICROPROTRUSION ARRAY AND METHODS OF MAKING A MICROPROTRUSION





**(57) Abstract:** A microprotrusion array is formed from a silicon wafer by a plurality of sequential plasma and wet isotropic and anisotropic etching steps. The resulting microprotrusions have sharpened tips or cutting edges formed by a wet isotropic etch.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# MICROPROTRUSION ARRAY AND METHODS OF MAKING A MICROPROTRUSION

## Field of the Invention

The present invention is directed to microdevice and to a microprotrusion array. The invention is also directed to methods of manufacturing a microdevice, and particularly microprotrusion arrays, for delivering or withdrawing a substance through the skin of a patient.

## Background of the Invention

Various microdevices are known in the art for performing different tasks. One application that has received interest in recent years is in the field of fluid control devices and microvalves. The microvalves have been shown to be useful in many industrial applications including the field of drug delivery, fuel delivery systems for internal combustion engines, as well as ink jet printers. These devices have been made by a number of different processes.

The many techniques that are commonly used in the fabrication of electronic devices and integrated circuit chips are suitable for micromachining of micromechanical devices. These microdevices are typically referred to as micro-electrical mechanical systems (MEMS). The devices are extremely small and can be made from numerous kinds of materials. A common material is silicon in the form of silicon wafers as used in the integrated circuit industry. Other materials that can be used include glass and ceramics.

An example of a microvalve is disclosed in U.S. Patent No. 6,056,269 to Johnson et al. The microvalve disclosed therein includes a silicon diaphragm with a valve seat and a flow channel. The diaphragm is positioned so it is able close against the valve seat when the diaphragm is deflected. The diaphragm is formed by etching and machining the silicon body. A separate actuating force is applied to the diaphragm to open and close the valve. The actuating device can be a pressurized fluid or a solenoid mechanism to apply a force to one side of the diaphragm.

Solenoid actuation of a valve in a gas chromatography assembly is known as disclosed in U.S. Patent No. 4,582,624 to Terry et al. The valve structure is formed by etching and machining a valve body to form the desired shapes. The solenoid

actuated devices are expensive to produce and portions of the device can not be manufactured efficiently.

Another area of microdevices is in the field of devices for delivering or sampling substances through the skin of a patient. The microdevices are typically small gauge needles or needle-like devices for penetrating the skin to the desired depth. Microneedle devices are desirable for many applications since they are able to deliver or withdraw a substance through the skin with minimal pain or discomfort to the patient.

One type of device that has gained attention is the microdevice that is able to penetrate the outer layers of the skin with less pain or discomfort than a standard cannula. These microdevices typically have needles that are a few microns to several hundred microns in length. The microdevices for delivering drugs through the skin form micro pores or cuts through the stratum corneum. By penetrating the stratum corneum and delivering the drug to the skin in or below the stratum corneum, many drugs can be effectively administered. The devices for penetrating the stratum corneum generally include a plurality of micron size needles or blades having a length to penetrate the stratum corneum without passing completely through the epidermis. Examples of these devices are disclosed in U.S. Patent No. 5,879,326 to Godshall et al.; U.S. Patent No. 5,250,023 to Lee et al.; WO 97/48440; and 6,334,856 to Allen et al.

Various methods have been used to produce the microneedles and microblades of the prior drug delivery devices. However, these methods have limited the shape and dimensions of the microneedle or microblade. Accordingly, there is a continuing needed in the industry for improved methods of forming microdevices.

## Summary of the Invention

The present invention is directed to microdevices, and particularly microprotrusions. The present invention is also directed to processes for forming microdevices.

Accordingly, a primary object of the invention is to provide a process for forming microdevices, and particularly microprotrusion arrays, in an efficient and economical manner.

Another object of the invention is to provide a process for forming microprotrusion arrays for delivering or withdrawing a substance through the skin of a patient.

Still another object of the invention is to provide a process for forming a microprotrusion array by wet etching of a silicon wafer.

A further object of the invention is to provide a process for forming a microprotrusion array by etching a substrate to produce microprotrusions having a substantially pyramid shape.

Another object of the invention is to provide an etching process using a wet isotropic etchant to form a conical shaped microprotrusion.

A further object of the invention is to provide a process for forming a microprotrusion array having an axial passage through the microprotrusions.

Still another object of the invention is to provide an etching process for forming an axial passage in a microprotrusion after forming the microprotrusion.

A further object of the invention is to provide a process for forming a microprotrusion by etching a substrate to form microprotrusions having an annular body and a conical shaped tip.

Another object of the invention is to provide a process for forming an annular microtube having a continuous sharp ridge on an annular face.

A further object of the invention is to provide a process of forming an oxide layer on a microprotrusion tip and removing the oxide layer to form a sharpened tip.

Still another object of the invention is to provide a process for forming a microprotrusion array by forming a ring-shaped masking layer on a substrate and etching the substrate to form annular microprotrusions.

A further object of the invention is to provide an etching process for forming microprotrusions having a beveled tip.

Another object of the invention is to provide a process for forming a microprotrusion array having microprotrusions with an outer annular face, where the outer annular face has a plurality of sharpened points.

These and other objects of the invention are basically attained by providing a monolithic microprotrusion device comprising: a base having a width and a length, a top face and a bottom face; a plurality of microprotrusions oriented in an array and extending from the top face of the base and being integrally formed with the base,

each of the microprotrusions having at least one face extending to an outer end thereof.

The objects of the invention are further attained by providing a method of forming a microprotrusion array comprising the steps of: providing a substrate having a top surface and a bottom surface, forming a patterned masking layer in a plurality of areas on the top surface, the areas of the patterned masking layer having a predetermined dimension corresponding to a microprotrusion and defining an exposed area of the top surface of the substrate around the areas of the masking layer, etching the exposed area of the top surface of the substrate to a sufficient depth to form a plurality of microprotrusions, and removing the masking layer from the substrate to expose the microprotrusions.

The objects of the invention are still further attained by providing a method of forming a microprotrusion array comprising the steps of providing a silicon substrate having a top surface with a masking layer, removing a portion of the masking layer to form a plurality of substantially ring shaped portions of the masking layer on the top surface, etching the top surface of the silicon substrate to form a channel extending through a center of each of the ring shaped portions, and to form an annular column around each of the ring shaped portions, and removing the ring shaped portions of the masking layer to form a microprotrusion array.

The objects of the invention are further attained by providing a method of forming a microprotrusion array comprising the steps of: providing a substrate having a top surface and a bottom surface; forming a plurality of recesses in the top surface, the recesses defining inclined surfaces with respect to a plane of the top surface, the inclined surfaces forming an array of sharpened tips, and removing a portion of the substrate around the sharpened tips to form an array of microprotrusions having an outer end formed by the sharpened tips.

The objects of the invention are still further attained by providing a method of forming a microprotrusion array, comprising the steps of providing a substrate having a top surface and a bottom surface, forming an array of microprotrusions on the top surface of the substrate, the microprotrusions having a top surface, and forming at least one bevel on the top surface of each of the microprotrusions.

The objects, advantages and other salient features of the invention will become apparent to one skilled in the art in view of the following detailed description

of the invention in conjunction with the annexed drawings, which form a part of this original disclosure.

**Brief Description of the Drawings**

The following is a brief description of the drawings that form a part of this original disclosure, in which:

Figure 1 is a side view of the microprotrusion array in a first embodiment of the invention;

Figure 2 is a perspective view of the microprotrusion array of the embodiment of Figure 1;

Figure 3 is a top view of the microprotrusion array of Figure 2;

Figures 4A-4H show the sequential steps in forming a microprotrusion array in a second embodiment of the invention;

Figures 5A-5I show the sequential steps in forming a microprotrusion array in a third embodiment of the invention;

Figures 6A-6M show the sequential steps in forming a microprotrusion array in a fourth embodiment of the invention;

Figures 7A-7J show the sequential steps in forming a microprotrusion array in a fifth embodiment of the invention;

Figures 8A-8L show the sequential steps in forming a microprotrusion array in a sixth embodiment of the invention;

Figures 9A-9O show the sequential steps in forming a microprotrusion array in a seventh embodiment of the invention; and

Figure 10 is a perspective view of the microprotrusion in an eighth embodiment of the invention showing multiple sharp tips on the annular face.

**Detailed Description of the Invention**

The present invention is directed to microprotrusion devices and to methods of forming the microprotrusion devices. More particularly, the invention is directed to methods of producing monolithic microprotrusion arrays and microdevices from the microprotrusion arrays.

The microdevices of the invention in preferred embodiments are microprotrusion arrays for penetrating the skin of a patient to a selected depth for delivering or withdrawing a substance through the skin of a patient. The term

"microprotrusion" as used herein refers to a structure that can penetrate one or more layers of the skin of a patient to a desired depth. The microprotrusions in preferred embodiments of the invention have a sharpened tip and can be conical or pyramid shaped. Microprotrusions, as the term is used herein, include projecting members having a square or circular cross-section with or without points or sharpened tips or cutting or scraping edges as in some embodiments discussed herein. The microprotrusions also encompass annular shaped columns having an axial passage.

Figures 1-3 show one embodiment of the microdevice 10 of the invention. Microdevice 10 includes a base 12 and a plurality of microprotrusions 14. In this embodiment, microprotrusions 14 are spaced apart in substantially uniformly spaced apart rows and columns. Microprotrusions 14 have a substantially pyramid shape with each side 16 having a concave surface. Sides 16 converge to a tip 18. Tip 18 of each microprotrusion 14 has a substantially flat top face 20 extending parallel to base 12. As shown in the top view of Figure 3, top face 20 has a substantially square shape.

Microdevice 10 includes a plurality of projecting members and is primarily used for penetrating the skin of a patient to a selected depth for delivering a substance to or withdrawing a substance from a patient. In preferred embodiments, microdevice 10 is coupled to a reservoir for delivering a substance and particularly a pharmaceutical composition to a patient. Alternatively, microdevice 10 includes a suitable extracting device, absorbent device or material for withdrawing and storing a fluid from a patient. Examples of substances that can be sampled from a patient include drugs, analytes and glucose.

The microprotrusions 14 of the invention have a length and width suitable for penetrating into the skin of a patient to a desired depth for delivering a substance where the substance can be absorbed and utilized by the body. The length of the microprotrusions 14 is generally determined by the substance being delivered and the delivery area on the skin. In embodiments of the invention, microprotrusions 14 can have a length of about 10 microns to about 2 mm. In one embodiment, microdevice 10 is a delivery device for penetrating the stratum corneum of the skin without piercing the stratum corneum to minimize pain and irritation to the patient. In this embodiment, microprotrusions can have a length of about 10 microns to about 100 microns, and generally about 10 microns to about 50 microns. Delivery and sampling devices intended to penetrate selected layers of the skin can have a length

of about 100 microns to about 1000 microns. In other embodiments, microdevice 10 is incorporated into a microabrader for cutting or scoring grooves in or through the stratum corneum to deliver or withdraw a substance through the skin.

Microdevice 10 can be made from various materials depending on the intended use and the substance being delivered or sampled. Suitable examples include metals, such as gold, polymeric materials, such as polycarbonates, and other non-metals. In preferred embodiments, microdevice 10 is made of silicon. Silicon is a preferred material since silicon wafers are readily available and can be machined and shaped using various known processes.

Microdevice 10 is preferably made from a silicon wafer by MEMS processing (microelectromechanical systems). Silicon is an element that exists in three forms, namely, crystalline, polycrystalline and amorphous. Silicon is an elastic and robust material that is particularly suitable for the microdevices of the invention. Ultrapure electronic grade silicon wafers that are available for the electronic industry are suitable for use in the invention.

Polycrystalline silicon, typically referred to as polysilicon, and amorphous silicon are generally deposited as thin films on a substrate with a typical thickness of less than 5 microns. Crystalline silicon substrates are commercially available as circular wafers. These silicon wafers are generally available in 100 mm diameters and about 150 mm diameters. The 100 mm diameter silicon wafers are about 525 microns thick.

The mechanical properties of the silicon wafers is dependent on the crystal orientation of the silicon. The crystal orientation determines the plane-selective etching of the silicon wafer as discussed hereinafter in greater detail. Silicon has a diamond lattice crystal structure that can be regarded as a simple cubic shape. The specific directions and planes within the crystal are designated in reference to the principal axes using a three integer notation enclosed in brackets, carets, parentheses and braces as known in the art. For example, [100] represents a specific vector direction of the cube, and <100> denotes the six directions. Similarly, (111) is a plane perpendicular to the [111] vector. The {111} vector represents all eight equivalent crystallographic planes. The angle between the {100} and {111} is important in micromachining processes since many etching processes selectively etch {100} planes, but not {111} planes as known in the art. Certain etching processes are able to etch different planes at different rates while others etch at

similar rates to control the shape of the finished article. Commercially available silicon wafers are predominantly {100} orientation indicating that the top surface is a {100} plane.

Microdevice 10 in preferred embodiments is made from a silicon wafer by various sequential masking and selective etching steps. In the embodiment of Figures 1-3, microdevice 10 is made by forming a patterned mask on the top surface of a silicon wafer. The mask is formed as an array of spaced-apart dots corresponding to the spacing and desired pattern of the microprotrusion array. In the embodiment of Figures 1-3, the dots have a substantially square shape to produce microprotrusions having substantially square tips. The dimensions of the dots of the mask material determine the final dimensions of the microprotrusions. In preferred embodiments, the mask is a photoresist material that is applied to the surface of the silicon wafer and exposed to a light source to pattern the photoresist layer.

After the photoresist is formed into the desired pattern, the exposed silicon is etched to remove a portion of the silicon. In the illustrated embodiment, the silicon is etched to form the inclined surface of the sides of the microprotrusions. This shape is typically obtained by a wet potassium hydroxide etching solution.

The microdevices of the invention are produced by various masking and etching processes to obtain different shaped microdevices. The mask in one embodiment is formed by photolithography to obtain the desired pattern of a photoresist. Alternatively, the mask can be formed from silicon nitride or silicon oxide layers. The etching step can be by wet etching, plasma etching, reactive ion etching (RIE) and deep reactive ion etching (DRIE).

Lithography is a process that is commonly used in MEMS fabrication processes that applies a photoresist layer on the substrate. The photoresist layer is a photosensitive emulsion layer that when exposed to light forms soluble and insoluble patterns. The photoresist layer is exposed to print an image of the mask onto the layer. The mask is a patterned opaque chromium layer on a transparent support such as glass. The mask defines the pattern of the finished photoresist layer.

After the photoresist layer is exposed to the selected pattern, the layer is immersed in an aqueous developer solution to dissolve the exposed portions and produce a latent image. A positive photoresist is an organic resin material containing a sensitizer. The photoresist is often spin coated on the silicon wafer to a thickness

of about 0.5 microns to 10 microns. The sensitizer prevents dissolution of unexposed photoresist during immersion in the developer. Exposure to light in the range of 200 to 450 nm decomposes the sensitizer so that the exposed regions immediately begin to dissolve in the developer. In a negative photoresist, the unexposed areas dissolve in the developer and exposed areas remain.

The substrate and the coatings can be etched by various etching processes. The etching process used determines the final shape of the microdevice so that the etching process is selected accordingly. Factors considered in selecting the etching process include isotropy, etch medium and the selectivity of the etch to other materials.

Isotropic etchants etch the substrate in all directions at a substantially uniform rate and produce rounded cross-sectional features. In contrast, anisotropic etchants etch primarily in one direction. Typically, anisotropic etchants etch along a specific crystal plane of a silicon wafer. Some anisotropic etchants primarily etch along one crystal plane, and to a lesser extent along other planes to form various shapes. Anisotropic etchants are used to produce well defined trenches or cavities delineated by flat and well defined surfaces. The crystal plans, and thus, the plane of etching, are not necessarily perpendicular to the surface of the silicon wafer. Wet etchants can be isotropic or anisotropic and are generally preferred in the invention due to the cost and ease of handling.

Isotropic wet etchants used in the invention include hydrofluoric acid (HF) and a mixture of hydrofluoric acid, nitric acid ($HNO_3$) and acetic acid ($CH_3COOH$) often referred to as HNA. Nitric acid oxidizes silicon to silicon dioxide. The silicon dioxide is removed by the hydrofluoric acid. The etch rate of silicon can vary from 1 micron/minute to 5 microns/minute by adjusting the proportion of the acids in the mixture.

Wet anisotropic etchants include metal hydroxides, such as sodium hydroxide, potassium hydroxide, cesium hydroxide, and the like. Other suitable hydroxides include ammonium hydroxide and tetramethylammonium ($(CH_3)_4NOH$). Another suitable anisotropic etch is an aqueous mixture of ethylene diamine and pyrocatechol (EDP).

Potassium hydroxide is the most commonly used anisotropic etchant. Potassium hydroxides etch silicon along the {111} planes at a rate 100 times slower than it etches {100} planes. This enables potassium hydroxide to effectively etch

silicon wafers to form V-shaped grooves and trenches that are precisely delineated by {111} crystallographic planes. The etch rate of potassium hydroxide on silicon is about 0.5 microns to 2 microns per minutes depending on the temperature and concentration of the etchant.

Etching of silicon using aqueous anisotropic etchants produce three-dimensional faceted structures formed by intersecting crystallographic planes. The design of the masking pattern determines the overall shape of the resulting structure.

The desired shape of the finished device is determined in part by the crystal orientation of the silicon wafer. For example, {100}-oriented wafers are typically used to form V-shaped cavities. The etch front begins at the opening of the mask and proceeds in the vertical direction (<100> direction) to produce a cavity with a flat bottom and slanted sides. The etch ultimately is self-limiting on the four intersecting planes to form an inverted pyramid or a V-shaped trough.

Concave corners bounded by {111} planes remain intact during etching. However, convex corners are immediately attacked by the etchant since any slight erosion of the convex corner exposes planes other than {111}. In this manner, a convex corner in the mask layout will be undercut during the etch so that the etch front will proceed underneath the mask.

A common dry etching process is a plasma-phase etch. Plasma etching basically involves forming chemically reactive neutrals and ions under the effect of an electric or magnetic field and accelerating the particles toward the target. Reactive species include $SF_6$, $CF_4$, $Cl_2$, $CClF_3$ and $NF_3$. One type of plasma etching is generally referred to as reactive ion etching (RIE). Inductively coupled plasma reactive ion etching (ICP-RIE) uses an externally applied RF magnetic field. Deep reactive ion etching (DRIE) is a high speed etching capable of anisotropically etching silicon.

The masking materials of the invention are selected according to the etching process being used to form the finished product. For example, silicon nitride is an excellent masking material against etching in potassium hydroxide. Silicon dioxide can be used as a masking layer for short etches in potassium hydroxide. Photoresist materials are readily etched in alkaline solutions and are not suitable for masking silicon etches. Silicon dioxide and silicon nitride are essentially unetched by tetramethyl ammonium hydroxide and ethylenediamine pyrocatecol, and thus, are good masking materials for these etchants.

Silicon dioxide layers on the silicon can be produced by oxidizing the silicon in dry oxygen or in steam at temperatures of 850-1150°C. Silicon dioxide layers as well as silicon nitride layers can be formed by chemical vapor disposition (CVD) at atmospheric pressure, at low pressure (LPCVD) or plasma-enhanced (PECVD). Silicon nitride ($Si_3N_4$) is often deposited at atmospheric pressure by reacting silane ($SiH_4$) and ammonia ($NH_3$) at about 700° to 900°C.

Silicon dioxide and silicon nitride can be etched selectively by various etchants. For example, hydrofluoric acid is a suitable wet etchant for silicon dioxide but not silicon nitride. Phosphoric acid ($H_3PO_4$) is a suitable etchant for silicon nitride but not silicon dioxide. Plasma etching can also be used to selectively etch the silicon dioxide or silicon nitride by selecting the etching gases. For example, oxygen and $CHF_3$ can be used to plasma etch silicon dioxide. Silicon nitride can be etched using $SF_6$.

In the following embodiments, the processes of the invention are discussed in reference to the drawings. The drawings schematically illustrate the forming steps and show the resulting structures in cross-section. It will be appreciated that where the drawings show a single microprotrusion, the actual device will contain a plurality of microprotrusions arranged in an array. The various masking materials and etchants as used in the process are selected to obtain the desired etching along the appropriate planes of the silicon substrate. The silicon substrate is typically a silicon wafer that is selected having the crystal planes oriented to obtain the desired shape of the microprotrusions. The various etching steps are also selected to etch the substrate to a desired depth to attain the desired result. Typically, the finished microprotrusions have a length of about 50 to 1000 microns and a width of about 50 to about 200 microns.

**Embodiment of Figures 4A-4H**

In this embodiment, a silicon wafer 24 is etched by various steps shown in Figure 4A-4G to produce a microprotrusion array 22 shown in Figure 4H. Referring to Figure 4A, a silicon wafer 24 having a top surface 26 and a bottom surface 28 is used to form the array. A silicon oxide layer 30 is formed on the top surface 26 and a silicon oxide layer 32 is formed on bottom surface 28 of silicon wafer 24. Silicon oxide layers 32 and 30 can be formed by various processes such as low pressure chemical vapor deposition. A photoresist layer is formed on silicon oxide layer 32

and is developed to form a plurality of spaced apart circular open areas 34 in silicon oxide layer 32. The open areas 34 correspond to the dimensions of an internal passage for each microprotrusion and are spaced apart a distance corresponding to the desired spacing of the microprotrusions of the finished product.

The open area 34 of silicon oxide layer 32 exposes a portion of silicon substrate 24 as shown in Figure 4B. A silicon nitride layer 36 is formed on the lower silicon oxide layer 30 and a silicon nitride layer 38 is formed on the upper silicon oxide layer 32 and open portion 34 as shown in Figure 4C. Silicon nitride layers 36 and 38 can be formed by various processes including low pressure chemical vapor deposition.

A photoresist layer is formed on silicon nitride layer 36 and developed to form a photoresist mask on silicon nitride layer 36 opposite open area 34. The dimensions of the photoresist layer correspond to the desired shape and size of the finished microprotrusion. The exposed portions of silicon nitride layer 36 and silicon oxide layer 30 are etched by a plasma etch to produce the nitride mask 40 surrounding the exposed surface 42 of silicon substrate 24 as shown in Figure 4D.

A wet isotropic etching agent is applied to surface 42 of silicon substrate 24 to etch silicon substrate 24 and oxide layer 30 to produce the microprotrusion 44 having the structure shown in Figure 4E. The nitride layers 40 and 38 are removed by a wet nitride etch as shown in Figure 4F. At this stage, silicon oxide layer 32 defines exposed area 34 on the underside of silicon substrate 24.

A plasma etch is applied to the underside of silicon substrate 24 and exposed area 34 to form an axial passage 46 through microprotrusion 44 as shown in Figure 4G. The plasma etch is applied in a manner to form a substantially smooth walled channel extending perpendicular to the plane of silicon substrate 24. As shown in Figure 4G, passage 46 is etched from the bottom surface of silicon substrate 24 and extends to silicon oxide layer 30. Silicon oxide layer 30 and 32 are removed by a silicon oxide etch to obtain the microprotrusion array 22 as shown in Figure 4H. Preferably, the silicon oxide layers are removed by a wet etching process.

This embodiment is able to produce a plurality of microprotrusions 44 simultaneously in a silicon substrate by forming the masking layers in selected locations on silicon substrate 24. The shape and length of microprotrusion 44 is determined by the etching process, the shape and dimensions of the masking layers and the time period that the etchant is applied. In the illustrated embodiment, the

silicon nitride masking layer 40 has a substantially circular shape which produces a conical shaped microprotrusion 44. In alternative embodiments, the silicon nitride mask 40 can have a square shape so that the etching step produces a pyramid shape microprotrusion.

**Embodiment of Figures 5A-5I**

In this embodiment, a microprotrusion array 48 shown in Figure 5I is formed from a silicon substrate 50 as shown in Figures 5A-5H. Referring to Figure 5A, silicon substrate 50 has a top surface 52 and a bottom surface 54. A silicon nitride layer 56 is deposited on top surface 52 of silicon substrate 50. A photoresist layer is applied onto silicon nitride layer 56 and exposed and developed to form a silicon nitride mask 58 on top surface 52 of silicon substrate 50 as shown in Figure 5B. Silicon nitride mask 58 has a shape and dimension corresponding to the desired shape of the microprotrusion tip. Referring to Figure 5C, an anisotropic wet etch is applied to top surface 52 of silicon substrate 50 to etch silicon substrate 50 along the inclined planes to form a conical shaped portion 60. Typically, the anisotropic etchant is potassium hydroxide which etches along the inclined planes as shown in Figure 5C.

The silicon nitride mask 58 is removed by a nitride etch resulting in the conical shaped projection 62 extending from silicon substrate 50 as shown in Figure 5D. A silicon oxide layer 64 is applied to the upper surface of silicon substrate 50 completely covering conical projection 62 and the etched surface 66. A photoresist layer is applied to oxide layer 64. The photoresist layer is exposed and developed to form a photoresist mask overlying incline surface 60 and exposing the portions of oxide layer 64 overlying etched surface 66 and the tip 68 of conical projection 62. The oxide layer 64 is then selectively etched to remove the exposed portions of the oxide layer. The photoresist mask is removed which results in the oxide mask 70 on the inclined surface 60 of projection member 62. As shown in Figure 5F, frustoconical shaped oxide mask 70 remains on incline surface 60 of projection member 62.

Oxide layer 70 exposes top surface 66 of silicon substrate 50 and tip 68. A photoresist layer is then applied over oxide layer 70, tip 68 and etched surface 66. The photoresist layer is then exposed and developed to form photoresist mask 72 as

shown in Figure 5G. Photoresist mask 72 substantially covers oxide layer 70 and surface 66 of substrate 50 and exposes tip 68.

An axial passage 74 is etched through substrate 50 from the top surface to form a continuous channel extending through conical projection 62 to bottom surface 54 of substrate 50. Photoresist mask 72 is removed to expose surface 66 as shown in Figure 5H. Preferably, axial passage 74 is formed by a plasma etch to form axial passage 74 along planes substantially perpendicular to bottom surface 54 of silicon substrate 50.

A plasma etch is then applied to surface 66 to etch along a plane perpendicular to bottom surface 54 and form a substantially annular face 76 around conical surface 60 as shown in Figure 5I. As shown in Figure 5I, the resulting microprotrusion 48 extends perpendicular from substrate 50 and includes a substantially cylindrical side wall 76 terminating in a conical upper face 60 and a sharp tip 78. Axial passage 74 extends from tip 78 to bottom surface 54.

**Embodiment of Figures 6A-6M**

In this embodiment, a microprotrusion array is formed from a silicon substrate 80 having a top surface 82 and a bottom surface 84. An oxide layer 86 is formed on top surface 82 of silicon substrate 80. A photoresist layer is formed on oxide layer 86. The photoresist layer is patterned and developed to form a substantially circular shaped photoresist mask 88 for each microprotrusion as shown in Figure 6B. Photoresist mask 88 defines a central exposed area 90 and a surrounding exposed area 92 of oxide layer 86. Exposed areas 90 and 92 of oxide layer 86 are removed by etching, such as by a plasma etch. The oxide etching results in an annular shaped oxide mask 94 on top surface 82 of silicon substrate 80.

Photoresist mask 88 is removed to expose oxide mask 94 as shown in Figure 6C. Oxide mask 94 defines an inner surface 96 and a surrounding surface 97 of upper surface 82 of silicon substrate 80. An isotropic etch is applied to surfaces 96 and 98 to form curved faces 98 extending from an etched surface 100 up to oxide mask 94. As shown in Figure 6D, the isotropic etch etches a portion of silicon substrate 80 underneath oxide mask 94 to define a raised portion 102 below oxide mask 94. Preferably, the isotropic etch is a wet etch as known in the art.

Oxide mask 94 is removed by an oxide etch. Preferably, the oxide etch is a wet etch using, for example, hydrofluoric acid. The oxide etch removes oxide mask

94 to exposed raised portions 102 defined by curved face 98 and a top face 104. As shown in Figure 6E, top face 104 of raised portion 102 has a substantially flat surface corresponding to original top surface 82. Raised portion 102 has a substantially annular shape extending upwardly from silicon substrate 80.

An oxide layer 106 is then formed on the top face of silicon body 80. Oxide layer 106 can be formed using various processes as known in the art. In one embodiment, the top face of silicon body 80 is heated in an oxygen containing atmosphere. Preferably, oxide layer 106 is formed in a manner to consume a portion of the outer surface of silicon body 80. Silicon oxide layer 106 is formed to a thickness such that the opposing curved surfaces 97 of raised portion 102 are consumed and merge to form a sharpened tip 108 as shown in Figure 6F.

A photoresist layer is applied onto the formed oxide layer 106. The photoresist layer is exposed and developed to form a photoresist mask 110 having an annular shape and overlying the sharpened tips 108. The exposed areas of oxide layer 106 are selectively etched and photoresist mask 110 removed. This results in an oxide mask 112 overlying sharpened tips 108 as shown in Figures 6G and 6H. As shown in Figure 6H, oxide mask 112 defines an inner surface 114 and an outer surface 116 of silicon substrate 80.

A masking material 118 is applied to surface 116 of silicon substrate 80 leaving center portion 114 exposed. An anisotropic etch, such as a plasma etch, is applied to center portion 114 to etch a substantially cylindrical shaped recess 120 in silicon substrate 80 surrounded by oxide mask 112 as shown in Figure 6I. Masking material 118 is then removed to expose a top surface 122 of silicon substrate surrounding oxide mask 112 as shown in Figure 6J.

An anisotropic etch, such as a plasma etch, is then applied to the top surface to top surface 122 and to etch recess 120 completely through silicon substrate 80 and form an axial channel 124. The anisotropic etch also etches top surface 122 to form an annular shaped microprotrusion 126 having a substantially cylindrical wall 128 extending perpendicular from an etched top surface 130. Oxide mask 112 is then removed by selective etching to expose the sharpened tip 108. As shown in Figures 6L and 6M, the resulting microprotrusion 126 extends upwardly from substrate 80. Sharpened tip 108 in this embodiment extends completely around a top face of microprotrusion 126 to form a continuous ridge.

**Embodiment of Figures 7A-7J**

In this embodiment, a microprotrusion array is formed from a silicon substrate 134 having a top surface 136 and a bottom surface 138. A nitride layer 140 is formed on top surface 136. An oxide layer 142 is formed by a low temperature forming process to cover nitride layer 140 as shown in Figure 7A. A photoresist layer is formed on oxide layer 142 which is then exposed and developed to form a photoresist mask. In this embodiment, the photoresist mask has an annular shape conforming to the transverse dimensions of the microprotrusion to be produced. The exposed areas of the nitride layer 142 and oxide layer 140 are etched to form an annular shaped mask 144. As shown in Figure 7B, mask 144 defines a central area 146 and an outer area 148 of silicon substrate 134.

A photoresist layer is applied to exposed surface 148 and mask 144 as shown in Figure 7C. Typically, photoresist layer 150 is applied as a continuous layer which is then exposed and developed to produce the mask. As shown in Figure 7C, central portion 146 of silicon substrate 134 is exposed and not covered by photoresist layer 150. An anisotropic etch such as a plasma etch is applied to the top surface to etch central portion 146 and form a cylindrical shaped passage 152 completely through silicon substrate 134 as shown in Figure 7D. Preferably, passage 152 is formed by a plasma etch to etch along the planes substantially perpendicular to top surface 136 of silicon substrate 134.

Photoresist mask 150 is removed as shown in Figure 7E followed by a second anisotropic etch to etch the exposed surfaces of top surface 136 of silicon substrate 134. As shown in Figure 7F, the anisotropic etch is carried out to form annular columns 154 having substantially cylindrical side walls 156 extending perpendicular to bottom surface 138 of silicon substrate 134.

Referring to Figure 7G, an oxide layer 158 is formed on the exposed surfaces of silicon substrate 134. As shown, oxide layer 158 covers outer face 156 of column 154 and the inner surface of channel 152. A wet nitride etch is applied to etch a portion of the exposed nitride layer of mask 144 as shown in Figure 7H. The nitride etch etches away a portion around the edge of nitride layer of mask 144 to expose a portion of the upper surface of annular column 154. Since the nitride etch attacks the inner and outer edges of the nitride layer of mask 144, the resulting nitride layer has a dimension less than the dimension of the top surface of annular column 154.

An isotropic etch is then applied to the exposed silicon around the mask 144 to form sharpened tips 160 having concave shaped surfaces 162. The remaining oxide layer 158 and nitride layer 140 are removed by a selective etch or stripping process as known in the art. The resulting microprotrusions 163 as shown in Figures 7J are defined by an annular column 158 having an annular shaped sharpened tip 160 extending upwardly from column 158.

**Embodiment of Figures 8A-8L**

In this embodiment, a microprotrusion array is formed from a silicon substrate 164 having a top surface 166 and a bottom surface 168. An oxide layer 170 is formed on top surface 166 as shown in Figure 8A. As in the previous embodiment, a photoresist layer is formed on oxide layer 170 and developed to produce a photoresist mask 172 having a substantially annular shape corresponding to the dimensions of the desired microprotrusion. Oxide layer 170 is selectively etched to produce an oxide mask 174 as shown in Figure 8B. Oxide mask 174 has an annular shape corresponding to the shape of photoresist mask 172. Photoresist mask 172 is then removed.

An isotropic etch is applied to the top surface 166 of silicon substrate 164 to form an etched surface defined by concave faces 176 below oxide mask 174. As shown in Figure 8C, the isotropic etch etches a portion of the silicon below mask 174 to form an annular shaped raised portion 178 having a dimension slightly less than the width of mask 174.

Referring to Figure 8D, a nitride layer 180 is deposited on the top surface of silicon substrate 164 and oxide mask 174. A photoresist mask 182 is formed on nitride layer 180. As shown in Figure 8E, photoresist mask 182 has an exposed layer overlying the central opening of oxide mask 174. An isotropic etch is applied on the top surface to etch an axial passage 184 through the exposed nitride layer 180 and through silicon substrate 164 to bottom surface 168. Preferably, the anisotropic etch is a plasma etch to etch along the plane substantially perpendicular to bottom surface 168 of silicon substrate 164. The photoresist layer 182 is removed as shown in Figure 8G. An anisotropic etch, which is preferably a plasma etch, is applied to the top surface to etch nitride layer 180 and silicon substrate 164 to form annular columns 186 as shown in Figure 8H. As in the previous embodiments, annular

column 186 is defined by a substantially cylindrical outer wall 188 extending perpendicular to bottom surface 168 of silicon substrate 164.

An oxide layer 190 is formed on the exposed surfaces of silicon substrate 164 to cover outer surfaces of annular column 186 and the inner surfaces of axial passage 184 as shown in Figure 8I. A nitride etch such as phosphoric acid is applied to remove the remaining portions of nitride layer 180 and to expose the concave surfaces 176 of raised portion 178 as shown in Figure 8J. An isotropic silicon etch is then applied to the exposed concave surfaces 176 as shown in Figure 8K to form a sharpened tip 192 on annular column 186. The remaining oxide layer 190 is then removed by an oxide etch such as hydrofluoric acid. The resulting microprotrusion 194 is defined by annular column 186 and annular tip 192 as shown in Figure 8L.

**Embodiment of Figures 9A-9O**

In this embodiment, a microprotrusion array is formed from a silicon substrate 196 having a top surface 198 and a bottom surface 200. As in the previous embodiments, an oxide layer 202 is formed on top surface 198 as shown in Figure 9A. A photoresist mask 204 is formed on oxide layer 202 to define the shape of the resulting microprotrusions. Photoresist mask 204 has a substantially annular shape and is formed from a photoresist layer that is exposed and developed as in the previous embodiments.

The exposed areas of oxide layer 202 are selectively etched, such as by a plasma etch, to form an annular shaped oxide mask 206. Photoresist mask 204 is removed and top surface 198 of silicon substrate 196 is etched with an isotropic etchant as shown in Figure 9C. The isotropic etchant produces an annular shaped raised portion 208 defined by concave outer faces 210 converging toward oxide mask 206. As in the previous embodiments, the isotropic etchant removes a portion of the silicon beneath oxide mask 206 as shown in Figure 9C.

Referring to Figure 9D, a nitride layer 212 is deposited on a top face to completely cover silicon substrate 196 and oxide mask 206. Referring to Figure 9E, a photoresist mask 214 is formed on nitride layer 212. Photoresist mask 214 overlies a small area of oxide mask 206. As shown in Figure 9E, photoresist mask 214 overlies an outer edge and covers a small arcuate portion of oxide mask 206. In preferred embodiments, photoresist mask 214 covers an arc of less than about one quarter of the circumference of oxide mask 206. Nitride layer 212 is then etched

selectively to form a nitride mask 216 conforming to the shape and dimensions of photoresist mask 214 as shown in Figures 9E and 9F. Preferably, the nitride etch is selective so that substantially no etching of the silicon occurs at this stage.

Photoresist mask 214 is then stripped to expose nitride mask 216 as shown in Figure 9G. Nitride mask 216 corresponds substantially to the dimensions of photoresist mask 214 and overlies a small portion of the outer edge of oxide mask 206 as shown in Figure 9G. In the embodiment illustrated, nitride mask 214 covers only a portion of the oxide mask. As discussed below, the dimensions of nitride mask 214 determine the final shape of the microprotrusions.

A photoresist mask 218 is applied over the top surface of silicon substrate 196, oxide mask 206 and nitride mask 216. A photoresist layer is applied, patterned, and developed to form photoresist mask 218 which exposes the top surface 219 of silicon substrate 196 surrounded by oxide mask 206 as shown in Figure 9H.

Referring to Figure 9I, an anisotropic etch is applied to the top surface to etch an axial passage 220 extending completely through silicon substrate 196 to bottom surface 200. Photoresist layer 218 is then removed as shown in Figure 9J. An anisotropic plasma etch is applied to form an annular shaped column 222 having a cylindrical side wall 224 as shown in Figure 9K. As shown in Figure 9K, the plasma etch etches the silicon substrate 196 along a plane substantially perpendicular to bottom wall 200. The plasma etch also removes a portion of the nitride mask 216 leaving a portion 226 of nitride mask 216 underlying oxide mask 206. An oxide layer 228 is then formed on the exposed surfaces of silicon substrate 196 and the oxide mask 206. As shown in Figure 9L, oxide layer 228 is not applied to nitride mask 216 so that nitride mask 216 is exposed. A nitride etch, such as phosphoric acid, is applied to remove the portion 226 of nitride layer thereby forming an opening to expose concave face 210 formed on annular column 222 as shown in Figure 9M.

An isotropic silicon etch is then applied to the exposed surface of annular column 222 through the opening formed by the nitride etch removing portion 226. The isotropic silicon etch etches the silicon along an interface extending away from the opening in the oxide layer 228 to form a curved etched outer face 230 as shown in Figure 9N. The silicon etch is applied for a sufficient time to etch the face 230 to form a sharpened tip 232. In one embodiment, the silicon etch is an isotropic etch. In further embodiments, the silicon etch can be a plasma etch, a wet isotropic etch or a wet anisotropic etch using potassium hydroxide.

Oxide layer 228 is then selectively removed by, for example, by hydrofluoric acid, to expose microprotrusions 234. Microprotrusions 234 have a generally cylindrical shape with an outer surface defined by the curved inner face 230 and sharpened tip 232.

**Embodiment of Figure 10**

In another embodiment of the invention, a microprotrusion 236 is formed having a substantially cylindrical side wall 238 and a base 240. As in the previous embodiments, microprotrusion 236 is formed from a silicon wafer by photolithography to form a circular shaped mask followed by anisotropic etching to form the microprotrusion with cylindrical side wall 238 and an axial passage. A mask is formed on the silicon wafer to define the dimensions of the microprotrusion and to produce sharpened tips 242. In one preferred embodiment, tips 242 are spaced apart around the circumference of microprotrusion 236. Each tip 242 is formed by concave faces such that a curved ridge 244 is formed between adjacent tips 242.

While several embodiments have been chosen to illustrate the invention, it will be understood by those skilled in the art that various changes and modifications can be made without departing from the scope of the invention as defined in the appended claims.

**WHAT IS CLAIMED IS:**

1. A method of forming a microprotrusion array comprising the steps of:

providing a substrate having a top surface and a bottom surface,

forming a patterned masking layer in a plurality of areas on said top surface, said areas of said patterned masking layer having a predetermined dimension corresponding to a microprotrusion and defining an exposed area of said top surface of said substrate around said areas of said masking layer,

etching said exposed area of said top surface of said substrate to a sufficient depth to form a plurality of microprotrusions, and

removing said masking layer from said substrate to expose said microprotrusions.

2. The method of claim 1, further comprising the step of forming an oxide layer on said top surface of said substrate.

3. The method of claim 2, wherein said patterned masking layer is a nitride layer.

24. The method of claim 1, wherein said substrate is made of silicon.

5. The method of claim 1, comprising the step of applying a patterned masking layer on said bottom surface of said base and etching a channel through said bottom surface to a top end of said microprotrusions.

6. The method of claim 3, further comprising the steps of

providing an oxide layer on said bottom surface of said substrate,

etching an area of said oxide layer on said bottom surface of said substrate oriented below said microprotrusion, and

etching a channel extending between said bottom surface to said top surface.

7. The method of claim 6, wherein prior to forming said nitride layer, said method comprises

etching a plurality of areas in said oxide layer on said bottom surface of said support, and

applying a second continuous nitride layer on said bottom surface to cover said oxide layer and etched area on said bottom surface.

8. The method of claim 6, wherein subsequent to said etching of said oxide layer on said bottom surface, said method comprises

etching said nitride layer an said bottom surface to expose said etched area in said oxide layer on said bottom surface, and

etching a channel through said substrate extending between said bottom surface to said top surface of said base.

9. The method of claim 1, further comprising the step of

forming an oxide layer on said top surface and said microprotrusions,

etching a portion of said oxide layer on said microprotrusions to expose a tip portion of said microprotrusions, and

etching said exposed tip portion of said microprotrusion to form a channel extending between said tip portion of said microprotrusion and said bottom surface of said base.

10. The method of claim 9, further comprising the step of applying a photoresist layer to said microprotrusions.

11. The method of claim 10, further comprising the step of etching said top surface around said photoresist layer on each of said microprotrusions to form an annular column.

12. A method of forming a microprotrusion array comprising the steps of

providing a silicon substrate having a top surface with a masking layer,

removing a portion of said masking layer to form a plurality of substantially ring shaped portions of said masking layer on said top surface,

etching said top surface of said silicon substrate to form a channel extending through a center of each of said ring shaped portions, and to form an annular column around each of said ring shaped portions, and

removing said ring shaped portions of said masking layer to form a microprotrusion array.

13. The method of claim 12, wherein said masking layer is an oxide layer on said substrate.

14. The method of claim 12, wherein prior to removing said ring shaped portions of said masking layer, said method comprises

etching said silicon substrate around said ring shaped oxide portions to form a pointed tip.

15. The method of claim 13, comprising

forming a plurality of ring shaped nitride portions on said oxide layer and etching said oxide layer around said ring shaped nitride portions to form said ring shaped portions from said oxide layer.

16. The method of claim 13, after removing said ring shaped oxide portions, said method further comprises the steps of

forming a second oxide layer on said microprotrusion array to sharpen said microprotrusions, and

removing said second oxide layer to exposed said sharpened microprotrusions.

17. The method of claim 16, comprising

applying a photoresist layer to said second oxide layer on said microprotrusions, and

etching said second oxide layer to form a ring shaped oxide layer prior to said step of etching said channels and annular portions.

18. The method of claim 13, said method further comprising

applying an oxide layer to an inner and an outer surface of said annular columns, and thereafter etching said silicon around said ring shaped oxide portions to form pointed tips on said columns.

19. The method of claim 18, further comprising applying a nitride layer onto said first oxide layer before etching said channels in said microprotrusions.

20. The method of claim 19, further comprising applying a photoresist layer onto said nitride layer and top surface of said substrate prior to forming said channels.

21. The method of claim 20, comprising the step of removing said photoresist layer prior to said step of forming said annular columns.

22. The method of claim 13, further comprising

applying a nitride layer to said top surface of said substrate and ring shaped portions prior to forming said annular columns,

applying a photoresist layer to said nitride layer overlying said ring shaped oxide portions, and

forming said channels, and thereafter forming said columns.

23. The method of claim 22, further comprising

forming an oxide layer on an outer surface of said column and an inner surface of said channel, and

removing said nitride layer from said columns, and further etching said silicon at an outer end of said columns to form a sharpened tip.

24. The method of claim 22, wherein prior to etching said silicon substrate, said method further comprises

forming an oxide layer on an outer surface of said columns and on an inner surface of said channel where a portion of a top end of said column is free of said oxide layer, and

etching said portion of said top end to said column to form a beveled tip.

25. The method of claim 12, further comprising cutting a beveled tip on said column.

26. The method of claim 25, further comprising providing a dicing saw, and cutting said beveled tip with said dicing saw.

27. The method of claim 25, wherein prior to cutting said bevel, said method further comprises encasing said column in a support medium.

28. The method of claim 27, wherein said support medium is a wax.

29. A method of forming a microprotrusion array comprising the steps of providing a substrate having a top surface and a bottom surface;

forming a plurality of recesses in said top surface, said recesses defining inclined surfaces with respect to a plane of said top surface, said inclined surfaces forming an array of sharpened tips, and

removing a portion of said substrate around said sharpened tips to form an array of microprotrusions having an outer end formed by said sharpened tips.

30. The method of claim 29, wherein said substrate is a material selected from the group consisting of silicon, silicon oxide, silicon nitride, epoxy resins, nickel and aluminum.

31. The method of claim 29, wherein said substrate is polysilicon or amorphous silicon.

32. The method of claim 29, wherein said inclined surfaces are formed by applying an anisotropic etchant to said top surface of said substrate and forming a beveled surface in a crystal plane of said substrate.

33. The method of claim 32, wherein said anisotropic etchant is selected from the group consisting of potassium hydroxide, ethylene diamine pyrocatecol, ammonium hydroxide, sodium hydroxide, CsOH, and $N_2H_4$.

34. The method of claim 29, wherein said inclined surfaces are formed by applying an isotropic etchant to said to surface of said substrate.

35. The method of claim 34, wherein said isotropic etchant comprises a mixture of hydrofluoric acid, acetic acid and nitric acid.

36. The method of claim 29, wherein said array of microprotrusions are formed by machining said top surface of said substrate around said sharpened tips.

37. A method of forming a microprotrusion array, comprising the steps of

providing a substrate having a top surface and a bottom surface,

forming an array of microprotrusions on said top surface of said substrate, said microprotrusions having a top surface, and

forming at least one bevel on said top surface of each of said microprotrusions.

38. The method of claim 37, wherein said microprotrusions have an outer surface, and said step of forming said bevel comprises

forming a non-oxidizing layer on said outer surface of said microprotrusions,

forming an oxide layer on said top surface of said microprotrusions,

etching said non-oxidizing layer to form an exposed area on said microprotrusions, and

etching said microprotrusions in said exposed area to form said bevel.

39. The method of claim 38, further comprising applying a non-etching layer on said non-oxidizing layer.

40. The method of claim 38, comprising removing said non-oxidizing layer.

41. The method of claim 37, wherein said microprotrusions are formed by selectively etching said substrate.

42. The method of claim 37, wherein said bevels are formed by selectively etching said substrate.

43. The method of claim 37, wherein said bevels are formed by selectively etching said microprotrusions.

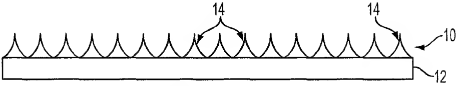



FIG. 1

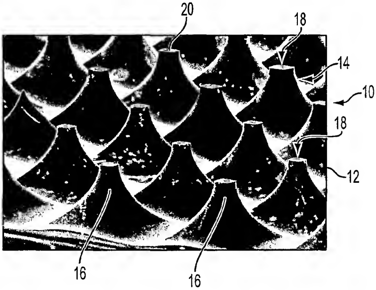



FIG. 2

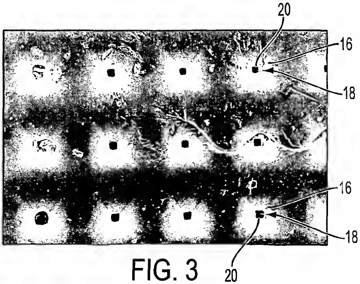



FIG. 3

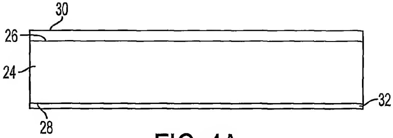



FIG. 4A

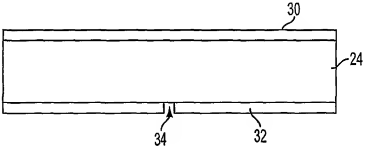



FIG. 4B

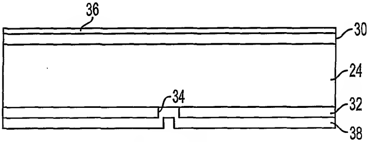



FIG. 4C

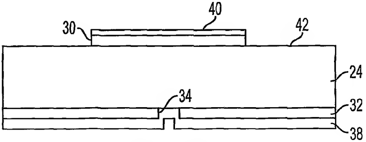



FIG. 4D

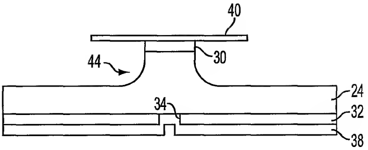



FIG. 4E

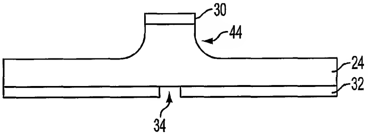



FIG. 4F

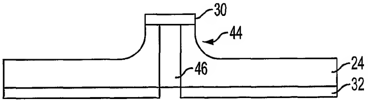



FIG. 4G

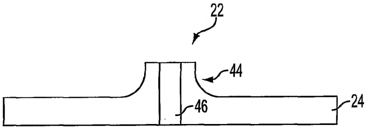



FIG. 4H

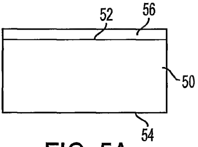



FIG. 5A

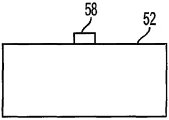



FIG. 5B

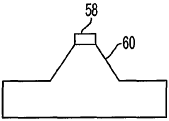



FIG. 5C

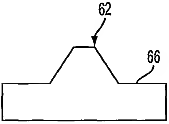



FIG. 5D

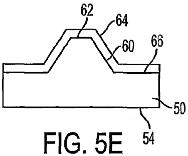



FIG. 5E

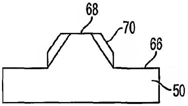



FIG. 5F

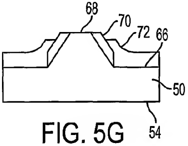



FIG. 5G

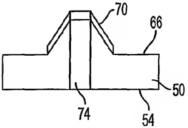



FIG. 5H

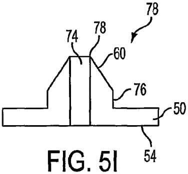



FIG. 5I

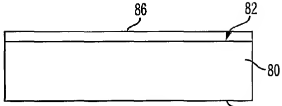



FIG. 6A

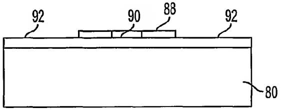



FIG. 6B

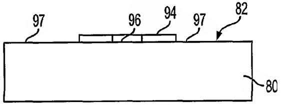



FIG. 6C

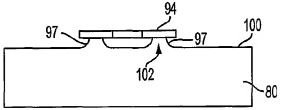



FIG. 6D

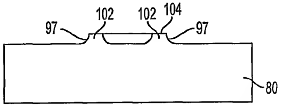



FIG. 6E

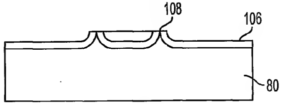



FIG. 6F

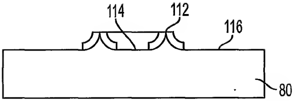



FIG. 6G

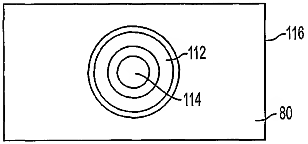



FIG. 6H

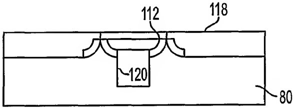



FIG. 6I

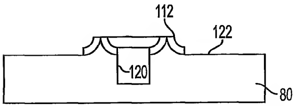



FIG. 6J

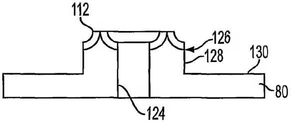



FIG. 6K

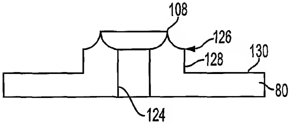



FIG. 6L

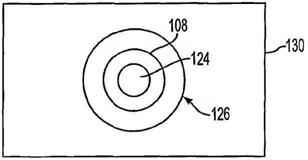



FIG. 6M

FIG. 7A

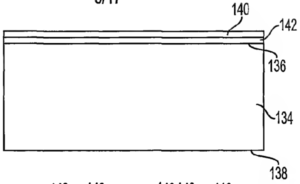



FIG. 7B

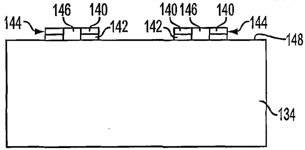



FIG. 7C

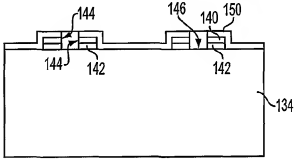



FIG. 7D

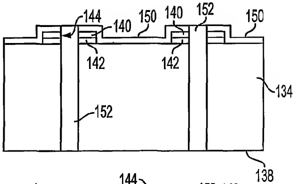



FIG. 7E

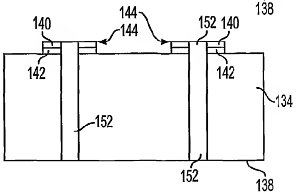


FIG. 7F

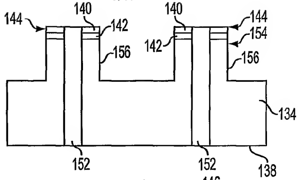



FIG. 7G

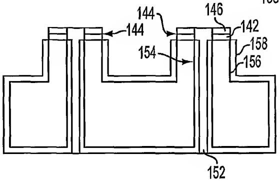



FIG. 7H

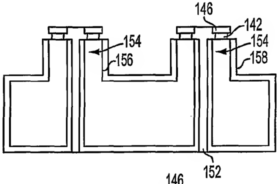



FIG. 7I

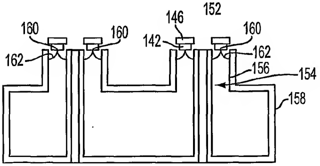



FIG. 7J

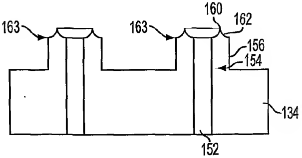


FIG. 8A

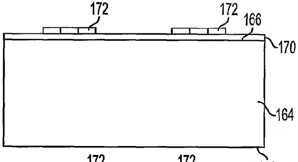



FIG. 8B

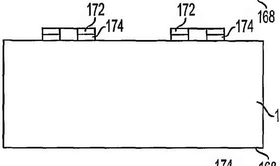



FIG. 8C

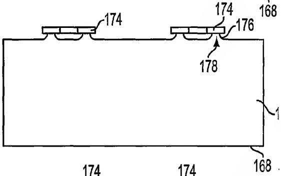



FIG. 8D

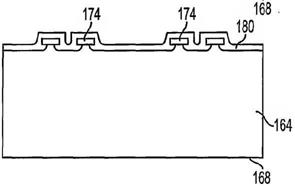
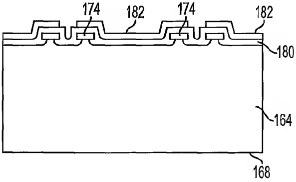



FIG. 8E



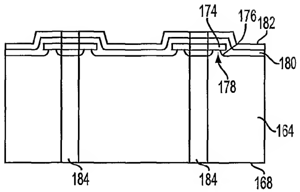



FIG. 8F

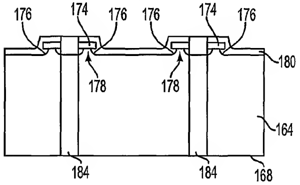



FIG. 8G

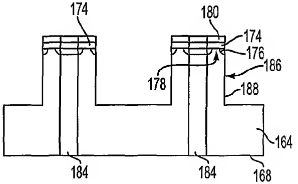



FIG. 8H

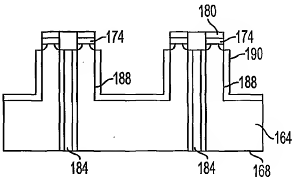



FIG. 8I

FIG. 8J

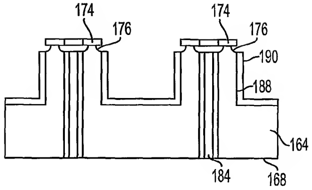



FIG. 8K

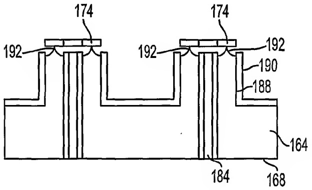
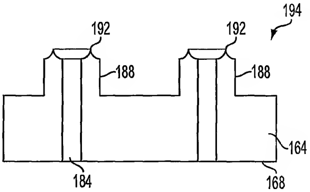



FIG. 8L



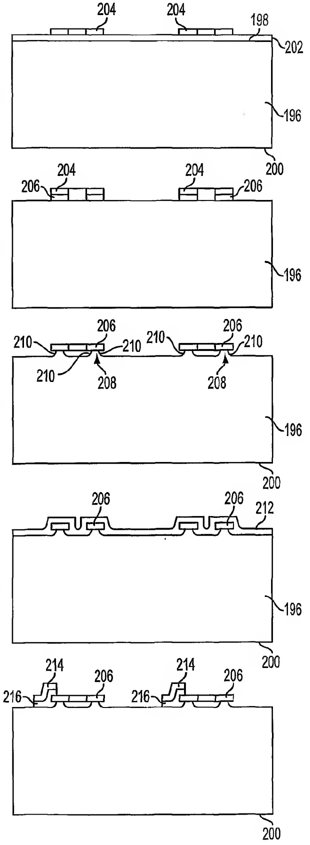


FIG. 9A
FIG. 9B
FIG. 9C
FIG. 9D
FIG. 9E

FIG. 9F

FIG. 9G

FIG. 9H

FIG. 9I

FIG. 9J

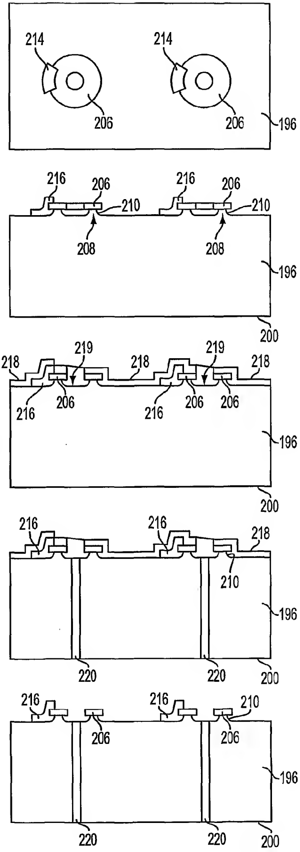


FIG. 9K

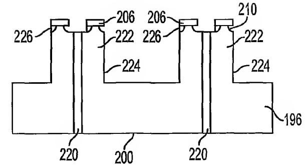



FIG. 9L

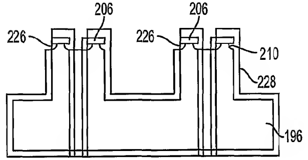



FIG. 9M

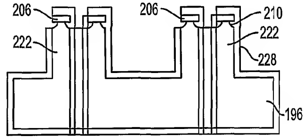



FIG. 9N

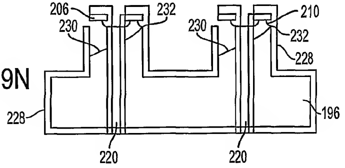
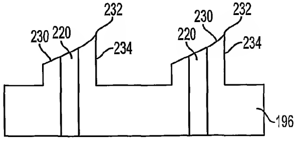



FIG. 9O



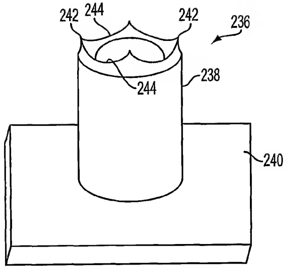



FIG. 10